ARKIV FÖR FYSIK Band 2 nr 46

Communicated 11 October 1950 by Einar Hammarsten and G. Borelius

A 180° mass spectrometer for biochemical research

By Hans von Ubisch

With 9 figures in the text

1. Introduction. This mass spectrometer is primarily intended for the isotopic analysis of light elements. However, some consideration has also been given to eventual future demands, which might include the chemical analysis of gaseous compounds. The experience gained from the 60° spectrometer in our laboratory (1) has been put to use in planning this spectrometer.

The instrument at present is only provisorily installed and in operation. The intention is to remove it next year to a new laboratory in Norrbacka, where ample space will be available for more comfortable arrangements than could be established at present. In the following descriptions only subjects of general interest have been included.

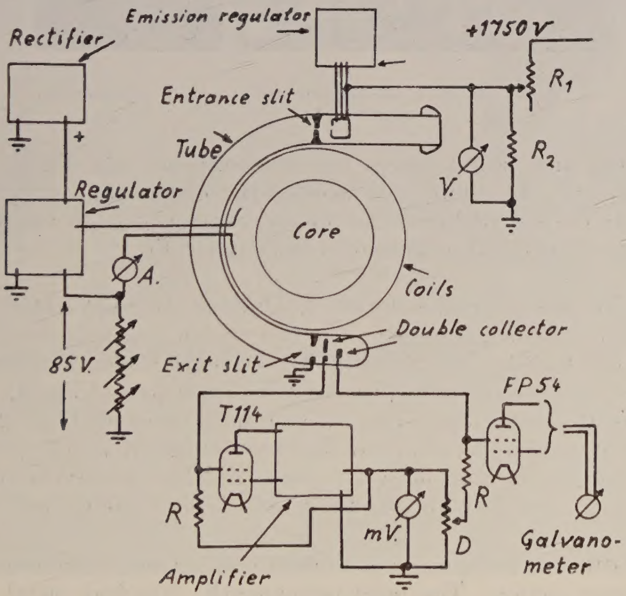

Fig. 1. Block scheme of mass spectrometer.

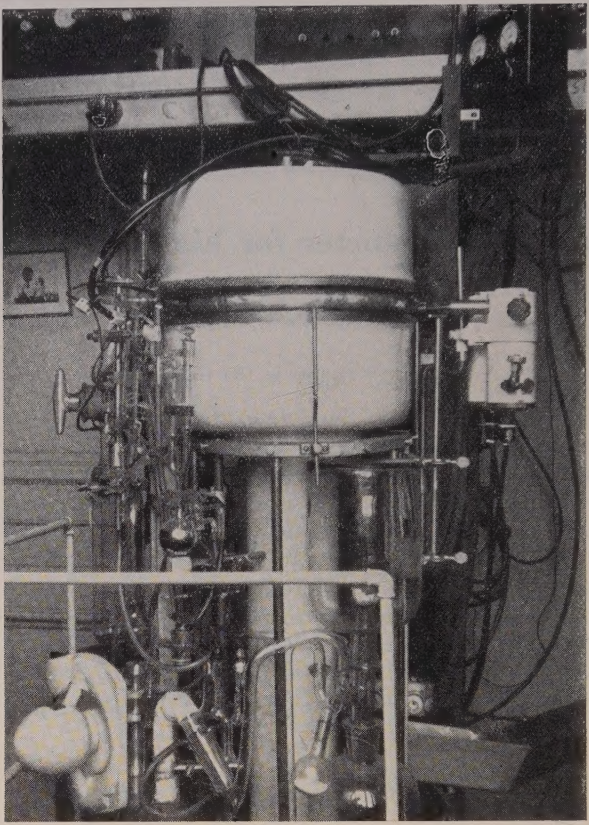

Fig. 2. Front view of magnet and vacuum equipment.

2. General lay-out. Fig. 1. gives the block-scheme and Figs. 2–3 two views of the spectrometer. The white pole-faces of the magnet appear in the center of these figures. At the left of Fig. 3 the present control panel is visible. Additional electrical equipment is placed in front on a balcony (cf. Fig. 2).

3. Magnet. The design is made by Dr. L. Dreyfus, ASEA, Västerås. The magnet has a cylindrical core which is forged. The two cup-shaped pole shoes are cast from extra soft steel (by Fagersta Bruks AB.). The coils are wound between watercooled copper flanges directly on the core (just visible in upper part of Fig. 3). Each consists of 3350 turns of 0.85 mm wire. The entire resistance is 169 Ω at 20° C. The assembled weight of the magnet is approximately 300 kg. It is supported by an iron column, with the gap in horizontal position at a height of 150 cm above the floor. This should be clear from the photographs. The cross section of the pole gap is 4×8 cm.

4. Analyzer tube. Several tubes have been built. They are all of pyrex and have an ellipsoidal cross section. The only permanently attached metal parts are the lead-in-wires. All the other parts fit elastically into the tube and can be removed

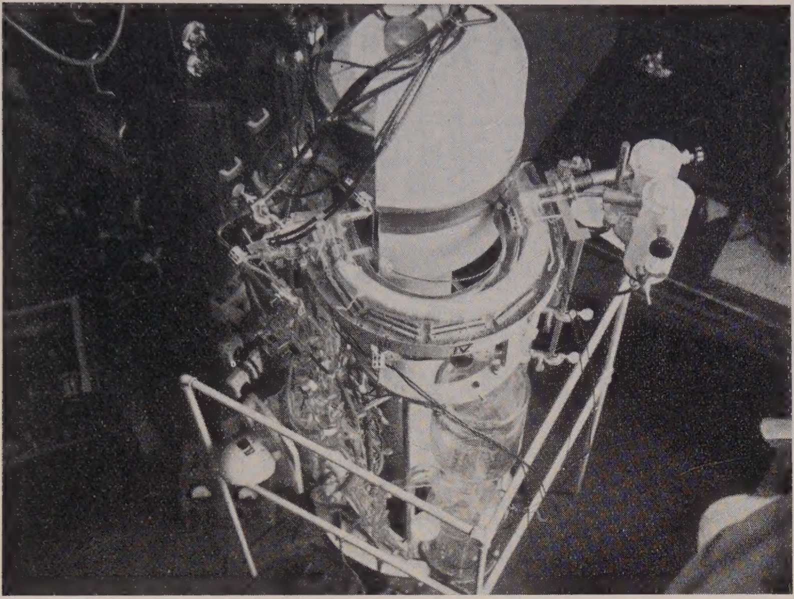

Fig. 3. Downward view with tube moved out and ready for baking.

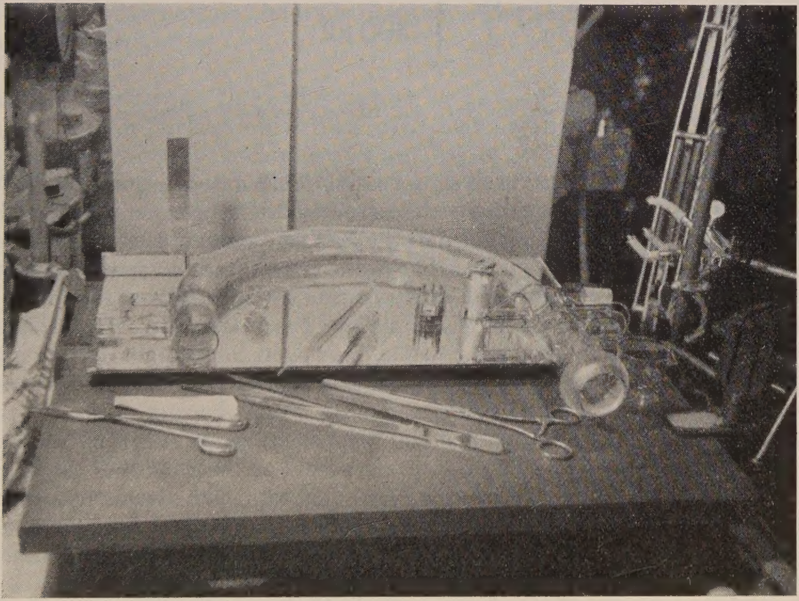

Fig. 4. Mechanical alignment of ion source and collectors by means of a mirror.

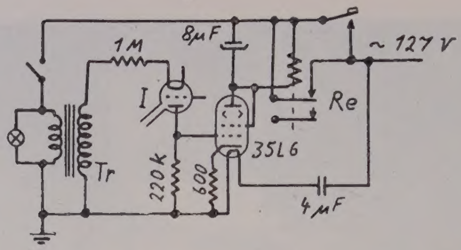

Fig. 5. High vacuum safety relay. I, ionization gauge (the cathode is kept cold). Tr, neon light transformer, smallest size. Re, relay with mercury switches.

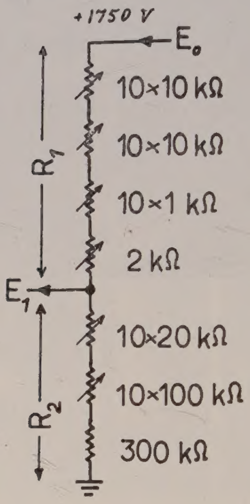

Fig. 6. Voltage divider. E_1, ion accelerating voltage.

and replaced. The ion source is thus accessible through the pumping line. The filament stems and the capillary leak are introduced through conical ground joints waxed with picien. Thus the filament and the leak are removable without necessitating the disconnection of other parts. The waxed joints are cooled by a few turns of a thin rubber hose carrying water. They cause no vacuum troubles.

Ion sources with and without deflector plates have been built. The plates should facilitate the establishment of a good focus. However, no definite advantage could be observed. The exit slit of the ion sources is 12 × 0.3 mm. The middle radius of the tubes is 17.5 cm.

The collectors are of the double type introduced by NIER (2). The openings are 12 × 12 mm and 14 × 1.5 mm. The resolution power of the tube is therefore approximately the same as that of the tube described by NIER. The mechanical alignments of the ion source and the collector were made by laying the tube upon a mirror (Fig. 4). Thus it is easy to observe visually any departure from perpendicularity to the mirror surface.

Most of the metal parts have been made of Nichrome V. However, the middle portion of a tube is shielded by a rectangular sheet of gold-plated copper gauze.

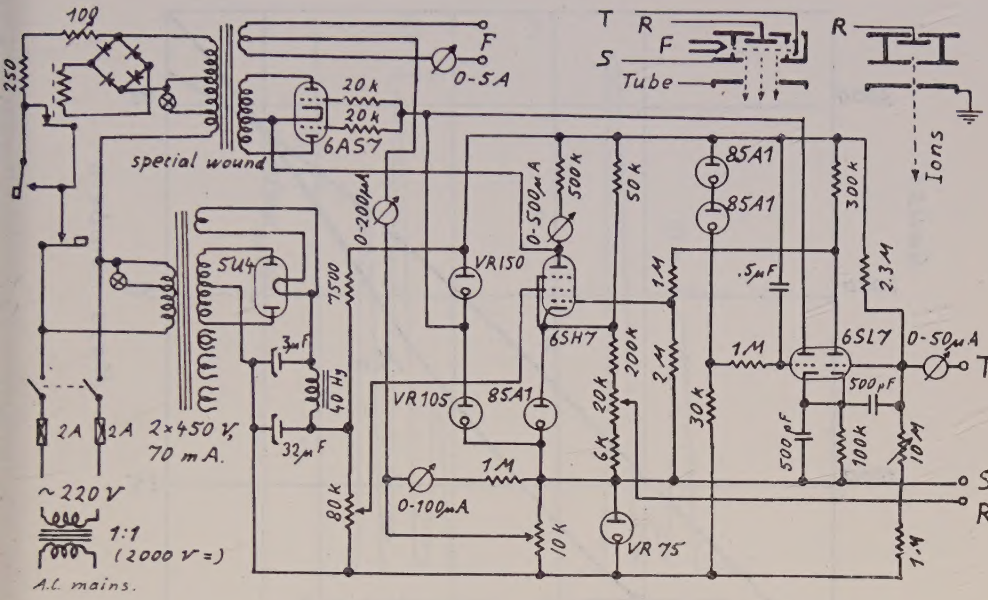

Fig. 7. Emission regulator.

If the wires in the sheet run at angles of 45° to the sides, the sheet can easily be rolled and bent and will fit snugly into the tube. In order to remove any electrostatic charges and leak currents on the walls of a tube, guard rings and lines of silver paste have been provided also.

5. Vacuum systems. In our laboratory we have had greater success with oil diffusion pumps than with the generally used mercury diffusion pumps. The design of the pumping system is partly clear from Fig. 2. It shows, from the right to the left, a forevacuum container built up of two glass flasks, a bottle containing "Dehydrite", a booster equipped with two small hot-wire manometers and a three-stage non-fractionating pump using Octoil S and air-cooled (lower part of figure), a cold trap using dry ice and alcohol and finally an ionization manometer (upper part of figure). The latter can serve also as the head of a safety relay (Fig. 5).

The gas inlet system may be seen in the middle part of Fig. 2, in front of the big stop-cock. It is evacuated by a pyrex mercury diffusion pump (not visible).

These vacuum systems and the analyzer tube are mounted on a horse-shoe shaped frame which can be turned by 90° around a vertical axis. In this way the tube can be moved out from the magnet, as is seen in Fig. 3. An oven can be placed on that frame right under the tube, as is also shown, and the tube baked in that position. The oven and the tube are covered during the baking by a hood not shown in this picture. It is possible to replace the assembled frame quickly with other equipment without breaking the vacuum.

6. Electrical equipment. (Fig. 1.) The regulator for the magnetizing current provides current stabilization. A reference voltage of 85 volts is provided by a

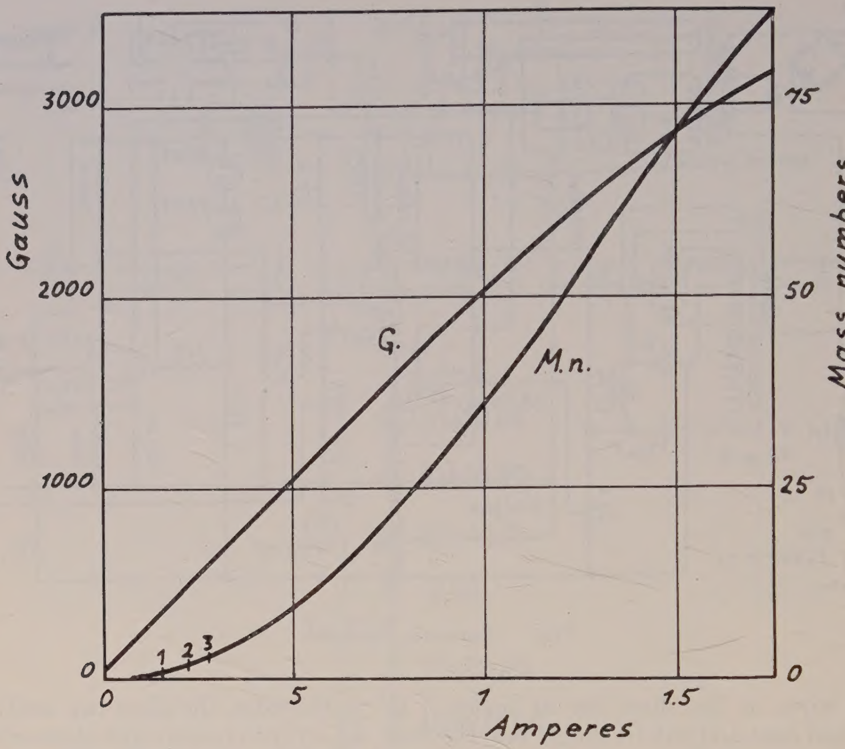

Fig. 8. Magnetization curve and curve for mass numbers. Ion accelerating voltage is 1750 V.

Philips voltage regulator valve Nr. 85 Al. However, the output voltage of the regulator can also be changed continuously by means of a small synchronous motor and a helipot potentiometer (not shown) if mass spectra are to be scanned.

The accelerating voltage is taken from the stabilized supply of the 60° spectrometer. The voltage divider used is shown schematically in Fig. 1 and in greater detail in Fig. 6. It permits the easy selection of any mass number and of intermediate half values within a sequence of 11 successive numbers arbitrarily chosen: The magnetic field is adjusted to focus the lowest mass number within the sequence, M, with $R_1 = 0$ and $E_1 = 1750$ V. R_2 is adjusted to equal M times 20 k Ω. If R_1 is increased then, by $n \times 20$ k Ω, the focus will be obtained for the mass number $M + n$. This follows from the principal equation of the mass spectrometer:

$$r_{\text{cm}} \cdot H_{\text{gauss}} = 144\sqrt{E_{1\,\text{volts}} \cdot M_{\text{units}}}$$

Fine adjustments and the scanning over a more limited range are carried out by means of the decade of 10×1 k Ω and the potentiometer of 2 k Ω. The setting of R_2 is not very critical.

The ion source supply circuit is shown in Fig. 7. Its special features are the use of the trap current, instead of the total emission, for the stabilization, and further improved stability against line voltage fluctuations.

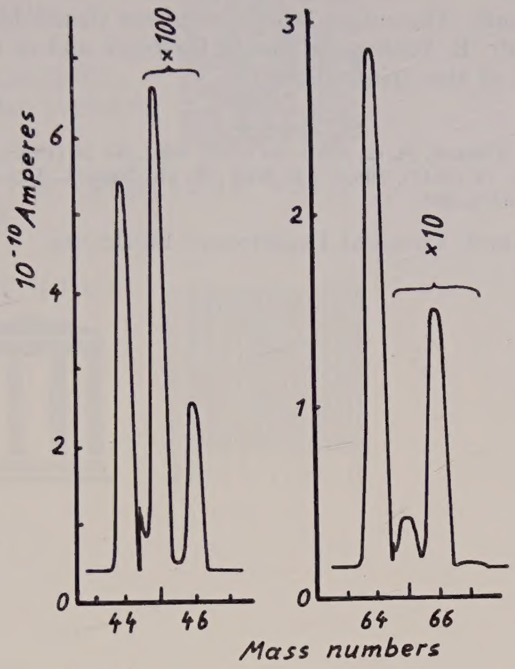

Fig. 9. Mass spectra of carbon dioxide and of sulphur dioxide.

The ion currents are measured by conventional BARTH—PENICK circuits. However, these were connected together as shown schematically in Fig. 1, and as is explained in an earlier paper by NIER, NEY and INGHRAM (3). The amplifier can also be connected to the right electrometer valve (Fig. 1) and the mass spectrometer can thus be used as a one-slit instrument with amplifier output. Provision is also made for carrying out absolute measurements of ion currents by means of a battery and the give-and-take decade unit D, i.e. by counterbalancing the voltage drop over any of the high-ohmic resistors R.

The electrometer valve circuits and the amplifier are fed from a stabilized rectifier. The electrometer valves are mounted in heavy-walled iron containers. Two high-ohmic resistors and a 3 M Ω resistor in each container are interchangeable by means of a switch.

7. Qualities. The magnetization curve and a curve for the mass numbers are shown in Fig. 8. Mass spectra of carbon dioxide and of sulphur dioxide are given in Fig. 9.[1] The spectrometer has already been used for work on deuterium, which is described in a following paper, and for relative isotopic abundance measurements of carbon in biochemical and mineralogic samples. The reproducibility of these measurements, in the present stage of the spectrometer, is within 0.1–0.2 %.

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[1] The shape of the peaks indicates some spherical aberration. Considerable improvement of the focus has, however, recently been achieved by the operator, Ingeniör Ryhage.

Acknowledgements. The author wishes to express thanks for the valuable technical assistance of Mr. E. Broberg, of Mr. F. Hagbjörk and of Ingeniör R. Ryhage in the construction of this spectrometer.

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Tryckt den 18 januari 1951

Uppsala 1951. Almqvist & Wiksells Boktryckeri AB